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METHODS OF TEST FOR MINERAL GYPSUM

(Second Revision)

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NEW DELHI 110002

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Indian Standard

METHODS OF TEST FOR MINERAL GYPSUM

(Second Revision)

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Indian Standard

METHODS OF TEST FOR MINERAL GYPSUM

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 21 May 1982, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 During the last few years, the consumption of gypsum in the manufacture of cement, fertilizers, insecticides, plaster of Paris, etc, has increased steadily. The major source of supply is from Rajasthan It is also mined in Madras, Uttar Pradesh and to a smaller extent in Gujarat, Jammu and Kashmir, and Maharashtra.

0.3 Fertilizer and cement are the two important industries in which gypsum finds use. High purity gypsum is utilized in large quantities in the manufacture of ammonium sulphate fertilizer. Gypsum of lesser purity in crushed condition is utilized in portland cement manufacture, where it acts as a retarder, controlling the setting time of cement.

0.4 Gypsum finds use in agriculture as a surface plaster for conserving moisture in soil and for aiding nitrogen absorption. Calcined gypsum finds use in plasters and manufacture of plaster of Paris. It is also used in the manufacture of partition blocks, sheets and tiles, plaster and insulating boards, and for stucco and lattice works. Ground gypsum, as filler, is utilized in paint, paper, rubber, textiles, etc. Gypsum blocks are used as building stones. In pottery, gypsum is used for moulding purposes. Besides, gypsum rock is used as flux in the smelting of nickel ores and in tin plate industry for polishing plates.

0.5 Alabaster, a massive and granular variety of gypsum is employed for statuary while the silky fibrous variety known as satin-spar is employed for making ornamental articles. Salenite, a crystalline variety is used to a limited extent for gypsum plate in microscopes. Low grade gypsum finds use in the manufacture of gypsum wall boards.

0.6 As it is sometimes advisable to recalculate the results obtained in analysis in order that they may become more enlightening, a procedure for the recalculation is also suggested in Appendix A.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2 - 1960[*].

1. SCOPE

1.1 This standard prescribes methods of test for mineral gypsum. It covers the methods for determination of fineness, free water, combined water, carbon dioxide, silica and acid insolubles, iron and aluminium oxides, calcium oxides, magnesium oxides, sodium chloride, sulphur trioxide, and various forms of calcium sulphate.

1.1.1 The specifications of the material are covered under IS : 1290-1965[†] and IS : 4266-1967[‡].

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977[§]) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. DETERMINATION OF FINENESS

3.1 Quantity Required — The quantity of the material required for this test depends on the particle size of the material. If the particles are not more than 6 mm in diameter, 100 g of the material shall be sufficient for this test; and if the largest particles are more than 25 mm in diameter, 1 000 g of the material shall be required. With these limitations, the quantity of the material required for this test may be left to the discretion of the operator.

3.1.1 It is, however, difficult to sieve very fine powder of the material and water cannot be used as washing agent without introducing errors due to hydration or solution. In such cases, *iso*propyl alcohol (99 percent) shall be used as washing agent and the procedure given under **3.3** shall be adopted.

3.2 Procedure for Coarse Powders — Determine fineness by sieving a known mass of the material dried at 45 ± 1°C through sieves of the specified sizes. Shake the sample through the specified sieves with as little abrasion as possible. Weigh the material retained on each sieve and calculate the fineness expressed as percentage of the mass of material taken for the test.

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[*] Rules for rounding off numerical values (*revised*).

[†] Specification for mineral gypsum (*revised*).

[‡] Specification for hydrated calcium sulphate from marine brine.

[§] Specification for water for general laboratory use (*second revision*).

3.3 Procedure for Fine Powders — Place *iso*propyl alcohol (99 percent) in a vessel 3 to 5 cm larger in diameter than the specified sieve to a depth above 5 cm. Place about 50 g of the material, accurately weighed, on the specified sieve and lower into the alcohol until the material is thoroughly wetted. Lift the sieve out of the alcohol with a swirling motion, permitting the alcohol to drain through the sieve back into the vessel. Repeat the process at least 8 times until the alcohol passes freely through the sieve and the residue is essentially free from fines. Wash the residue with about 100 ml of clear alcohol and then blot the bottom of the sieve with a soft, dry and lint-free cloth. Dry the sieve with the residue at 45 ± 1°C. Shake the sieve for exactly 2 minutes. In case of dispute, a mechanical shaker shall be employed. Remove the residue with a camel hair brush to a tared sheet of glazed paper and weigh.

3.3.1 If desired, to permit simultaneous determination of coarser fractions, the residue after drying but before shaking may be transferred to a series of coarser sieves up to 1-mm IS Sieve nested in the order of sequence.

4. DETERMINATION OF FREE WATER

4.1 Procedure — Weigh accurately about 500 g of the material as received and spread it in a thin layer in a suitable vessel. Place in an oven and dry at 45 ± 1°C for 2 hours, then cool in an atmosphere free from moisture and weigh. Repeat till constant mass is obtained. The loss in mass corresponds to free water and shall be calculated as a percentage of the sample taken for the test.

4.2 Calculation

$$\text{Free water, percent by mass} = \frac{100\,(A - B)}{A}$$

where

A = mass in g of the material taken for the test, and

B = mass in g of the material after drying.

5. PREPARATION OF SAMPLE

5.1 Procedure — Weigh about 500 g of the material and spread it in a thin layer in a suitable vessel. Place in an oven and dry at 45 ± 1°C till constant mass is obtained, then cool in an atmosphere free from moisture. Grind the material, so that it passes through 250-micron IS Sieve taking extreme care not to expose the material unduly to moisture or overheating.

Thoroughly remix the ground sample, and store this prepared sample in an air-tight container for analysis.

6. DETERMINATION OF COMBINED WATER

6.1 Procedure — Place about 1 g of the *prepared sample* in a covered crucible which has been previously dried and weighed. Dry it in an oven at 215° to 230°C to constant mass. Calculate the loss in mass as percentage of the sample.

6.2 Calculation

$$\text{Combined water, percent by mass} = \frac{100\,(A - B)}{A}$$

where

A = mass in g of the prepared sample taken, and
B = mass in g of the material after drying.

7. DETERMINATION OF CARBON DIOXIDE (CO_2)

7.1 Apparatus—Apparatus consists of a 250-ml round bottom flask closed with rubber stopper or preferably standard glass joint with three holes for one tap funnel for addition of dilute hydrochloric acid, one water cooled reflux condenser and one tube for introduction of CO_2 free air. The top of the condenser is connected to a series of vessels *B*1 to *B*7 as follows:

*B*2, *B*3 and *B*4 are Nesbitt tubes (or Mid-vale tubes),
*B*1 and *B*7 are bubblers (Arnold bulb),
*B*5 is 125 ml bubbler, and
*B*6 is a Mid-vale tube.

The Arnold bulbs (*B*1 and *B*7) contain syrupy phosphoric acid and are meant to indicate the rate of flow of gas through the apparatus (1 to 2 bubbles per second) and *B*5 bubbler contains concentrated sulphuric acid to remove bulk of moisture from the gas. The acid level in the bulb *B*1, *B*5 and *B*7 should be just to cover the end of the delivery tube. *B*6 meant for removal of carbon dioxide from the incoming air, is packed with cotton wool to cover the end of the delivery tube and with soda asbestos to the level indicated in the figure. *B*2(a) and *B*2(b) are packed with soda asbestos covered with a 15-20 mm layer of anhydrous magnesium perchlorate *B*2(b) is meant for guard against any escape of CO_2 from *B*2(a). *B*3 for removal of hydrogen sulphide is packed with copper sulphate soaked in anhydrous magnesium perchlorate. *B*4 contains magnesium perchlorate only. In each Nesbitt tube the reagent should be kept in place with the plug of cotton wool. The tubes are connected together with a suitable rubber/polyethylene tubing. The supply of air through the apparatus is obtained from a filer pump or a small electric motor and is regulated with a screw clip. A spirit lamp or micro burner may be used to heat the flask.

7.2 Reagents

7.2.1 *Dilute Hydrochloric Acid* — 1 : 1 (*v*/*v*).

7.2.2 *Magnesium Perchlorate Anhydrous* — 710 micron IS Sieve.

7.2.3 *Soda Asbestos* — 140 mm IS Sieve.

7.2.4 *Pumice Impregnated With Copper Sulphate* — Anhydrous. Crush pumice to approximately 5·60 mm IS Sieve size, sift free from dust, and transfer 60 g to a casserole. Cover with a concentrated solution of 30 to 35 g of copper sulphate, evaporate to dryness while constantly stirring and then heat for 3 to 4 hours at 150° to 160°C in an air oven. Cool in a desiccator and preserve in a glass-stoppered bottle. Use freshly dried material in *B*3.

7.3 Procedure — Assemble the apparatus as shown in Fig. 1 with the bulbs packed as described. With all the taps fully open, start the pump pulling air through the apparatus and note that Arnold bulbs (*B*1 and *B*7) have air at about the same rate of 1 to 2 bulbs per second passing through them. Beginning at *B*7, close and then open each tap in turn and note that with each tap closed, the current of air through the Arnold bulb *B*1 ceases to flow. This will indicate that each tap is operating correctly. Now close all the taps and remove the Nesbitt tubes *B*2(a) and *B*2(b) to a balance case and allow to stand for 30 minutes. Carefully wipe the outside of the bulb with a clean cloth and weigh.

7.3.1 Weigh 1 g for Grade 1 and 5 g for Grade 2 of the sample into the flask, moisten with a little water and transfer about 50 ml of diluted hydrochloric acid to the tap funnel. Reassemble the apparatus as shown in the Fig. 1 with taps 1 to 7 open once more and with a clip between the suction pump and Arnold bulb *B*1 restricting the flow of air through the apparatus fully closed.

7.3.2 Start the pump and open the screw clip to produce a steady stream of bubbles through Arnold bulbs. Open the tap of the funnel to admit hydrochloric acid slowly to the flask. Close the tap of the funnel before all the acid has been added.

7.3.3 When all evolution of carbon dioxide has apparently ceased, boil the solution gently for about 10 minutes, turn off the burner and keep the air current flowing for a further period of 30 minutes. Close all the taps. Remove the Nesbitt tubes *B*2(a) and *B*2(b) to a balance case, allow to stand as before, wipe the tubes and weigh. Increase in mass is due to absorbed carbon dioxide. Run reagent blank through all the steps without taking the sample. This should be deducted from the mass of carbon dioxide determined above.

7.4 Calculation

$$\text{Carbon dioxide, percent by mass} = \frac{M_1 - B_1}{M}$$

where

M_1 = mass in g of Nesbitt tubes $B2$(a) and $B2$(b), after the test;

B_1 = mass in g of Nesbitt tube $B2$(a) and $B2$(b) for blank experiment; and

M = mass in g of the material taken for the test.

8. DETERMINATION OF SILICA AND ACID INSOLUBLES

8.1 Reagents

8.1.1 *Dilute Hydrochloric Acid* — 1 : 5 (*v/v*).

8.1.2 *Concentrated Hydrochloric Acid* — *see* IS : 265 - 1976*.

8.2 Procedure — Place about 1 g of the prepared sample, accurately weighed, in a porcelain dish. Add about 50 ml of dilute hydrochloric acid and evaporate on a low flame to almost dryness. Cool and add enough of concentrated hydrochloric acid to wet the residue thoroughly. Add about 10 ml of water and boil. Filter through filter paper (Whatman No. 30 or equivalent) and wash the residue with water. Preserve the residue on the filter paper and transfer the filtrate to the same porcelain dish. Evaporate the filtrate to dryness and heat at about 120°C for 1 hour and then cool. Add enough concentrated hydrochloric acid to wet thoroughly. Add about 25 ml of water, boil and filter again through filter paper (Whatman No. 30 or equivalent). Transfer both the filter papers containing the residue to the same tared crucible. Ignite and weigh. Preserve the filtrate and washings for the test described in **9**.

8.3 Calculation

$$\text{Silica and acid insolubles, percent by mass} = \frac{100\ A_1}{A}$$

where

A_1 = mass in g of the ignited residue, and

A = mass in g of the prepared sample taken for tne test.

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*Specification for hydrochloric acid (*second revision*).

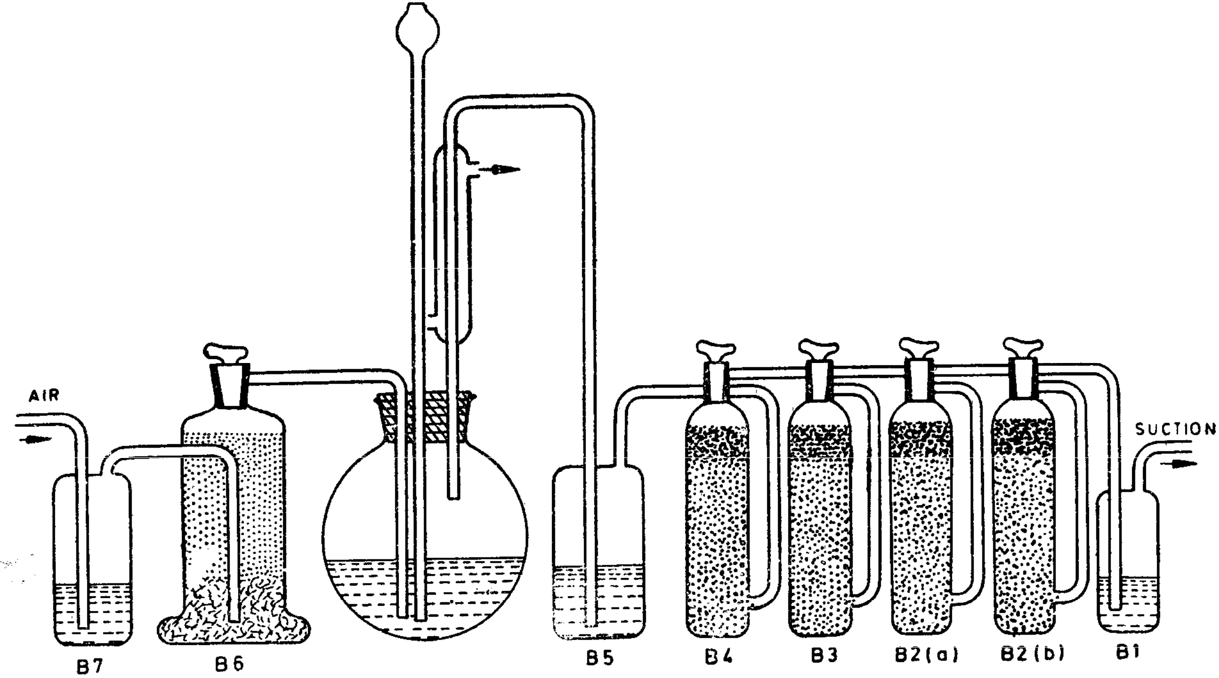

FIG. 1 APPARATUS FOR THE GRAVIMETRIC DETERMINATION OF CARBON DIOXIDE

9. DETERMINATION OF IRON AND ALUMINIUM OXIDES

9.1 Reagents

9.1.1 *Concentrated Nitric Acid — see* IS : 264-1976*.

9.1.2 *Ammonium Chloride* — solid.

9.1.3 *Methyl Red Indicator* — Dissolve 0·1 g of methyl red in 100 ml of rectified spirit (*see* IS : 323-1959†).

9.1.4 *Dilute Ammonium Hydroxide* — approximately 5 N.

9.1.5 *Ammonium Nitrate Solution* — one percent (*m/v*).

9.2 Procedure — To the filtrate reserved in **8.2**, add a few drops of concentrated nitric acid and boil to ensure oxidation of iron. Add about 4 g of ammonium chloride previously dissolved in water. Add two drops of methyl red indicator and make it just alkaline with dilute ammonium hydroxide solution. Boil for a few minutes until the precipitate coagulates. Filter through a filter paper. Wash the precipitate with hot ammonium nitrate solution until the washings are free from chlorides. Preserve the filtrate for test described in **10.** Dry the filter paper containing the residue and ignite to constant mass.

9.3 Calculation

$$\text{Iron and aluminium oxides, percent by mass} = \frac{100\,A_1}{A}$$

where

A_1 = mass in g of the ignited residue, and

A = mass in g of the prepared sample taken for the test in **8.2.**

10. DETERMINATION OF CALCIUM OXIDE

10.0 General — Calcium oxide may be determined either by the permanganate method or by the EDTA method. In case of dispute, the permanganate method shall be considered as the referee method.

10.1 Permanganate Method

10.1.1 *Reagents*

10.1.1.1 *Dilute hydrochloric acid* — approximately 5 N.

10.1.1.2 *Dilute ammonium hydroxide* — approximately 5 N and 1 N.

10.1.1.3 *Methyl red indicator* — same as in **9.1.3.**

---

*Specification for nitric acid (*second revision*).

†Specification for rectified spirit (*revised*).

10.1.1.4 *Oxalic acid solution* — saturated in water.

10.1.1.5 *Ammonium oxalate solution* — approximately 5 percent (*m*/*v*).

10.1.1.6 *Dilute potassium permanganate solution* — approximately 0·1 percent (*m*/*v*).

10.1.1.7 *Dilute sulphuric acid* — approximately 5 N.

10.1.1.8 *Standard potassium permanganate solution* — approximately 0·05 N.

10.1.2 *Procedure* — To the filtrate reserved in **9.2**, add dilute hydrochloric acid in slight excess. Heat to near boiling and add a few drops of ammonium hydroxide (5 N) to make it clearly alkaline. If necessary, add a few more drops of methyl red indicator. Add oxalic acid solution drop by drop until the indicator becomes just red. While keeping the solution near boiling; add about 40 ml of ammonium oxalate solution slowly with stirring. Allow the precipitate to stand on a boiling water-bath for 30 minutes and then decant the supernatant solution through a filter paper (Whatman No. 42 or equivalent). There is no need to transfer the precipitate to the filter paper. Dissolve the precipitate remaining in the beaker in minimum quantity of dilute hydrochloric acid by heating to boiling and add dilute ammonium hydroxide (5 N) dropwise until a precipitate begins to form. Dilute to about 100 ml, heat to near boiling and add about 20 ml of ammonium oxalate solution slowly with stirring. Allow to stand on a boiling water-bath for 2 hours.

10.1.2.1 Transfer the precipitate to the filter paper and wash the precipitate first with dilute ammonium hydroxide (1 N) and then with cold water until the filtrate does not decolourize a hot dilute potassium permanganate solution (0·1 percent) in presence of dilute sulphuric acid. Reserve both the filtrate washings for test in **11.1.2**.

10.1.2.2 Puncture the filter paper and transfer the precipitate to the beaker with a fine jet of hot water. Then wash the filter paper five times with hot dilute sulphuric acid using 10 ml of the acid each time and collecting the washings in the beaker. Heat the contents of the beaker to dissolve the precipitate. Allow the solution to cool and then dilute it in a graduated flask to 100 ml. Pipette out 25 ml of the solution into a conical flask, add 20 ml of dilute sulphuric acid and heat to 60°C. Titrate with standard potassium permanganate solution.

10.1.3 *Calculation*

$$\text{Calcium oxide (CaO), percent by mass} = \frac{11{\cdot}22\,NV}{A}$$

where

N = normality of the standard potassium permanganate solution,

V = volume in ml of standard potassium permanganate solution used in the titration, and

A = mass in g of the prepared sample taken for the test in **8.2**.

10.2 EDTA Method

10.2.1 *Reagents*

10.2.1.1 *Ammonium chloride — Ammonium hydroxide buffer solution* — Dissolve 67·5 g of ammonium chloride in 570 ml of concentrated ammonium hydroxide and make to one litre.

10.2.1.2 *Potassium hydroxide approximately 10 N*—The solution should be freshly prepared from AR quality potassium hydroxide 56 g dissolved in 100 ml water.

10.2.1.3 *Standard calcium chloride solution 0·01 N* — Dissolve 0·500 g of pure calcium carbonate (calcite crystals) in 10 ml of approximately 3 N hydrochloric acid and dilute to a volume of exact 1 litre.

10.2.1.4 *Eriochrome black T indicator* — Dissolve 0·5 g of Eriochrome black T (F 241) and 4·5 g of hydroxylamine hydrochloride in 100 ml of 95 percent ethanol.

10.2.1.5 *Patton and Reeder's indicator* — Mix 0·1 g of 2-hydroxy (2-hydroxy-4 sulphur-l-napthylaxo) 3-naphthoic acid with 10 g of potassium chloride and grind into a fine mixture in a glass mortar. Preserve in a well stoppered dry amber coloured glass bottle.

10.2.1.6 *Ethylenediaminetetraacetate (EDTA) (Versenate) solution approximately 0·01 N* — Dissolve 2·00 g of disodium dihydrogen ethylenediaminetetraacetate and 0·05 g of magnesium chloride hexahydrate in water and dilute to a volume of 1 litre. Standardize the solution against standard calcium chloride solution using the titration procedure given in **10.2.2.2**. The solution is standardized, using each of the indicators Eriochrome black *T*, and *P* and *R* indicator.

10.2.2 *Procedure*

10.2.2.1 Dilute the filtrate reserved in **9.2** to 500 ml in a graduated flask. Use this solution for **10.2.2.2** and **11.2.2**.

10.2.2.2 Pipette out 50 ml aliquot into two white porcelain dishes of 100 ml capacity. Dilute to about 50 ml with de-ionized water. Add about 50 mg each of potassium cyanide and hydroxylamine hydrochloride about 0 2 to 0·4 g, *P* and *R* indicator and quickly titrate till the colour changes to violet blue. Note the volume of EDTA consumed. Add the same volume of EDTA less 1·0 ml directly to the second dish. Add 5 ml of potassium hydroxide solution and about 50 mg each of potassium cyanide and hydroxylamine hydrochloride. Add 0·4 g indicator and immediately titrate with continuous stirring until no further colour changes and the test solution is clear blue without any tinge of violet.

10.2.3 *Calculation*

$$\text{Calcium oxide (CaO), percent by mass} = \frac{140{\cdot}2\ V_2 M_2}{A}$$

where

V_2 = volume in ml of standard EDTA solution used in the titration,

M_2 = molarity of EDTA solution, and

A = mass in g of the prepared sample taken for the test in **10.2.2.1**.

11. DETERMINATION OF MAGNESIUM OXIDE

11.0 General — Magnesium oxide may be determined either by pyrophosphate method or by EDTA method. In case of dispute, pyrophosphate method shall be considered as the referee method.

11.1 Pyrophosphate Method

11.1.1 *Reagents*

11.1.1.1 *Concentrated nitric acid* — same as in **9.1.1**.

11.1.1.2 *Concentrated hydrochloric acid* — same as in **8.1.2**.

11.1.1.3 *Diammonium hydrogen phosphate solution* — approximately 25 percent (*m/v*).

11.1.1.4 *Phenolphthalein indicator solution* — Dissolve 0·1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

11.1.1.5 *Dilute ammonium hydroxide* — same as in **10.1.1.2**.

11.1.2 *Procedure* — Evaporate the filtrate and washings collected in **10.1.2.1** to dryness. Add to the residue 30 ml of concentrated nitric acid and evaporate again to dryness. To the residue left, again add about 4 ml of concentrated nitric acid and heat to fuming on a sand bath. Cool and dissolve the residue in concentrated hydrochloric acid. Dilute with water to 100 ml, add 20 to 30 ml of diammonium hydrogen phosphate solution and 2 to 3 drops of phenolphthalein indicator. Heat to boiling and add dilute ammonium hydroxide (5 N) until a pink colour appears and slight precipitate forms. Stir for a minute and when the precipitate becomes distinctly crystalline add more dilute ammonium hydroxide (5 N) until deep colour is obtained. Set aside and allow the precipitate to stand overnight. Filter, wash the precipitate with dilute ammonium hydroxide (1 N) until washings are free from chlorides. Place the filter paper with its contents in a tared silica crucible. Dry in an air-oven, char the filter paper by gentle heating over low flame and then slowly increase the flame and finish by heating at 900° to 950°C till the precipitate is almost white. Cool the crucible in a desiccator and weigh.

11.1.3 *Calculation*

$$\text{Magnesium oxide, percent by mass} = \frac{36{\cdot}21\, M_1}{M}$$

where

M_1 = mass in g of the precipitate obtained, and

M = mass in g of the prepared sample taken for the test in **8.2**.

11.2 EDTA Method

11.2.1 *Reagents*

11.2.1.1 *Erichrome black T indicator* — Dissolve 0·5 g of the dye and 5 g of hydroxylamine hydrochloride in 100 ml of alcohol.

11.2.1.2 *Triethanolamine*

11.2.1.3 *Ammonium hydroxide-ammonium chloride buffer solution (pH 10)*– Dissolve 68 g of ammonium chloride in 200 ml of water, add 570 ml of concentrated ammonia (relative density 0·91) and dilute to 1 litre.

11.2.1.4 *Potassium cyanide* — 5 percent.

11.2.1.5 *Standard magnesium solution* — 0·02 M. Weigh 4·926 g of magnesium sulphate ($MgSO_4{\cdot}7H_2O$) and dissolve it in water. Make up the volume to one litre.

11.2.1.6 *Standard EDTA solution* — 0·02 M. Dissolve 7·44 g of disodium ethylene diamine tetra-acetate dihydrate in water and make up the volume to one litre and standardize it as follows:

> Take 10 ml of standard magnesium solution in a conical flask. Add 20 ml of water, 1 ml of eriochrome black T indicator and 25 ml of ammonium hydroxide-ammonium chloride buffer solution, and titrate with standard EDTA solution until the colour changes from wine-red to distinct blue.

$$\text{Molarity of EDTA solution} = \frac{10\, M_1}{V_1}$$

where

M_1 = molarity of standard magnesium solution, and

V_1 = volume in ml of EDTA solution used for titration.

11.2.2 *Procedure* — Pipette out an aliquot volume same as in calcium oxide determination (**10.2.2.2**) of the diluted solution, reserved in **10.2.2**, into a 250-ml conical flask. Add 20 ml of water and 50 ml of ammonium hydroxide-ammonium chloride buffer solution. A few drops of KCN, hydroxylamine hydrochloride and triethanolamine are added and then titrate with standard EDTA solution until the colour changes from wine-red to distinct blue.

11.2.3 *Calculation*

$$\text{Magnesium oxide (MgO), percent by mass} = \frac{100{\cdot}8\,(V_2 - V)\,M_2}{A}$$

where

V_2 = volume in ml of standard EDTA solution used in **11.2.2**,

V = volume in ml of standard EDTA solution used in **10.2.2.2**,

M_2 = molarity of standard EDTA solution as obtained in **11.2.1.4**, and

A = mass in g of the prepared sample taken for the test in **8.2**.

12. DETERMINATION OF SULPHUR TRIOXIDE AND CALCIUM SULPHATE DIHYDRATE

12.0 General — Sulphur trioxide and calcium sulphate contents are calculated from the mass of barium sulphate precipitate obtained from the sample.

12.1 Reagents

12.1.1 *Dilute Hydrochloric Acid* — same as in **8.1.1**.

12.1.2 *Barium Chloride Solution* — Dissolve about 12 g of barium chloride crystals in 100 ml of water.

12.1.3 *Concentrated Nitric Acid* — (*see* IS : 264-1976*).

12.1.4 *Concentrated Sulphuric Acid* — (*see* IS : 266-1977†).

12.2 Procedure — 0·2 g sample decomposed with 50 ml of dilute hydrochloric acid. Filter through No. 40 Whatman filter paper transferring the entire solution, wash with hot water 6 times or till free from sulphate.

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*Specification for nitric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

Dilute the filtrate to 200 ml. Boil the solution and precipitate the sulphate with this stream of 20 ml of barium chloride. Digest on water bath for 4 hours.

12.2.1 Filter through No. 42 Whatman Filter paper. Wash with hot water till free from chloride. Transfer into a 25-ml porcelain crucible. Burn the filter paper at low heat, then ignite at about 800°C. Treat the ignited precipitate with one or two drops each of concentrated nitric acid and sulphuric acid to moisten the precipitate and ignite. Cool in a desiccator and weigh.

12.3 Calculation

a) Sulphur trioxide (SO_3), percent by mass $= \frac{34{\cdot}30 \times M_1}{M}$, and

b) Calcium sulphate dihydrate ($CaSO_4{\cdot}2H_2O$) $= \frac{73{\cdot}74\, M_1}{M}$

where

M_1 = mass in g of the precipitate, and
M = mass in g of the prepared sample taken for the test.

13. ALTERNATE METHOD FOR DETERMINATION OF CALCIUM SULPHATE

13.1 Reagents

13.1.1 *Ammonium Acetate Solution* — Dissolve 454 g of ammonium acetate in 2 litres of water. Add ammonium hydroxide to make the solution distinctly ammoniacal.

13.1.2 *Diatomaceous Silica* — Wash Kieselguhr free from soluble matter and dry to constant mass at 105° to 110°C.

13.1.3 *Ammonium Hydroxide* — 20 percent (*m*/*m*).

13.1.4 *Ammonia Wash Solution* — Dilute 100 ml of ammonium hydroxide to one litre with water.

13.2 Procedure — Place about 4 g of the material, accurately weighed, in a 600-ml beaker. Add 350 ml of ammonium acetate solution, stirring the mixture thoroughly so as to loosen the matter from the bottom of the beaker. Add about 0·2 g of diatomaceous silica, accurately weighed, to the mixture and heat at 70°C on a water-bath for 30 minutes with frequent stirring. Keep the solvent distinctly ammoniacal during this period by adding ammonium hydroxide, if necessary. Filter the mixture through a weighed Gooch crucible applying suction, if necessary. Stir frequently and keep the diatomaceous silica suspended in the liquid. Wash the residue with five 10-ml portions of hot ammonium acetate solution, draining thoroughly after each washing. Repeat washing with eight 10-ml portions of ammonia wash solution. Dry the crucible in an oven at 70°C to constant mass.

13.3 Calculation

$$\text{Calcium sulphate } (CaSO_4.XH_2O), \text{ percent by mass} = \frac{A-(B-C)}{A}$$

where

A = mass in g of the material taken for the test,

B = final mass in g of the crucible and the contents after drying, and

C = initial mass in g of the crucible plus mass in g of diatomaceous silica used as filter-aid.

NOTE — This is only an approximate method and determines calcium sulphate in all its forms including water soluble impurities.

14. DETERMINATION OF SODIUM CHLORIDE

14.1 Apparatus

14.1.1 *Nessler Cylinder* — 50 ml capacity (*see* IS : 4161-1967*).

14.2 Reagents

14.2.1 *Dilute Nitric Acid* — approximately 5 N.

14.2.2 *Silver Nitrate Solution* — approximately 5 percent (m/v).

14.2.3 *Standard Sodium Chloride Solution* (*A*) — Dissolve 1 g of sodium chloride (previously dried at 100° to 105°C to constant mass), accurately weighed, in 1 000 ml of water in a graduated flask. Transfer 10 ml of the solution in another 1 000-ml flask and dilute the solution to mark. One millilitre of the solution contains 0·01 mg of sodium chloride.

14.2.4 *Standard Sodium Chloride Solution* (*B*) — Dissolve 0·5 g of sodium chloride (previously dried at 100° to 105°C to constant mass), accurately weighed, in water and make up the solution to 100 ml. Dilute 10 ml of the solution to 100 ml. One millilitre of the solution contains 0·5 mg of sodium chloride.

14.3 Procedure — Boil about 1 g of the prepared sample, accurately weighed, with 10 ml of water. Filter and wash with five 5-ml portions of hot water. Collect the filtrate and washings in Nessler cylinder. Add 5 ml of dilute nitric acid and 1 ml of silver nitrate solution. Dilute the solution with water to 50 ml. In the other Nessler cylinder, add 5 ml of dilute nitric acid and 1 ml of silver nitrate solution and then with the help of a burette, add standard sodium chloride solution *A* or *B* till the turbidity in the cylinder containing the material under test is the same as that in the control cylinder when diluted to 50 ml. Solution *A* to be used where the amount of sodium chloride does not exceed 0·01 percent. For larger percentage of sodium chloride, use solution *B*.

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*Specification for Nessler cylinders.

14.4 Calculation

$$\text{Sodium chloride (NaCl), percent by mass} = 0{\cdot}1 \times \frac{VM_1}{M}$$

where

V = volume in ml of standard sodium chloride solution added,

M_1 = mass in mg of sodium chloride in 1 ml of the standard solution used, and

M = mass in g of the prepared sample taken for the test.

APPENDIX A

(*Clause* 0.6)

PROCEDURE FOR RECALCULATION OF RESULTS

A-1. PROCEDURE

A-1.0 Since it is frequently advisable to recalculate the results obtained in the chemical analysis in order that they may be more enlightening, the following procedure is recommended.

A-1.1 Multiply percentage of magnesium oxide (*see* **11**) by 2·091 to find the percentage of magnesium carbonate.

A-1.2 Multiply percentage of magnesium oxide (*see* **11**) by 1·091 to find percentage of carbon dioxide present as magnesium carbonate.

A-1.3 Deduct carbon dioxide present as magnesium carbonate (*see* **A-1.2**) from the total carbon dioxide determined (*see* **7**) and multiply the difference by 2·274 to find percentage of calcium carbonate.

A-1.4 Add together the percentages of silicon dioxide, iron and aluminium oxides, magnesium carbonate and calcium carbonate and report the aggregate.

A-1.5 Multiply the percentage of calcium carbonate (*see* **A-1.3**) to find the percentage of calcium oxide present as calcium carbonate.

A-1.6 From the total percentage of calcium oxide (*see* **10**) deduct the percentage of calcium oxide present as calcium carbonate (*see* **A-1.5**). The remainder may be called ' available calcium oxide '.

A-1.7 The percentage of ' available calcium oxide ' (*see* **A-1.6**) should bear to the percentage of sulphur trioxide (*see* **12**) a ratio of 0·699 to 1.

A-1.8 If the ' available calcium oxide ' is in excess, multiply the percentage of sulphur trioxide by 0·699 and subtract the result from percentage of available calcium oxide. The remainder is reported as ' excess calcium oxide '.

A-1.9 If sulphur trioxide is in excess, multiply the percentage of ' available calcium oxide ' by 1·430 and subtract the result from the percentage of sulphur trioxide. The remainder is reported as ' excess sulphur trioxide '.

A-1.10 Add together the percentages of the ' available calcium oxide ' and sulphur trioxide and subtract the ' excess calcium oxide ' or ' excess sulphur trioxide '. The remainder is the percentage of total calcium sulphate.

A-1.11 If calcium sulphate is present as $CaSO_4.\frac{1}{2}H_2O$, the percentage of total calcium sulphate (*see* **A-1.10**) should bear to the percentage of combined water (*see* **6**) a ratio of 15 12 to 1. Determine which of the two is in excess.

A-1.12 If calcium sulphate is in excess and some of it is present in anhydrous form, multiply the percentage of combined water by 15·12 to find the percentage of calcium sulphate present as $CaSO_4.\frac{1}{2}H_2O$. The difference between the total calcium sulphate and the percentage of calcium sulphate as $CaSO_4.\frac{1}{2}H_2O$ is the calcium sulphate in anhydrous form.

A-1.13 If water is in excess, some of the calcium sulphate is present as gypsum ($CaSO_4.2H_2O$). Let X be the percentage of calcium sulphate hemihydrate, and Y be the percentage of dihydrated calcium sulphate:

then:

a) $X + Y =$ percentage of total calcium sulphate (as found in **A-1.10**) + percentage of water, and

b) $0{\cdot}062\,06\,X + 0{\cdot}209\,3Y =$ percentage of combined water.

Solve these equations for *X* and *Y*. Report *X* as percentage of ' Calcined gypsum ' ($CaSO_4.\frac{1}{2}H_2O$). Report *Y* as percentage of gypsum ($CaSO_4.2H_2O$).

A-2. CALCULATION

A-2.1 Having made the calculations, the results may be reported as follows:

Constituent	*Percent*
Gypsum ($CaSO_4.2H_2O$)	...
Calcined gypsum ($CaSO_4.\frac{1}{2}H_2O$)	...
Anhydride ($CaSO_4$)	...
Excess calcium oxide (CaO)	...
or	
Excess sulphur trioxide (SO_3)	...
Sodium chloride (NaCl)	...
Other ingredients	...
Total	100·00

NOTE — The presence of the different forms of calcium sulphate may be corroborated by a microscopic examination.

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